



TITLE:

Dipterexと五硫化リンとの反応

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CITATION:

西沢, 吉彦 ...[et al]. Dipterexと五硫化リンとの反応. 防虫科学 1960, 25(4): 132-133

ISSUE DATE:

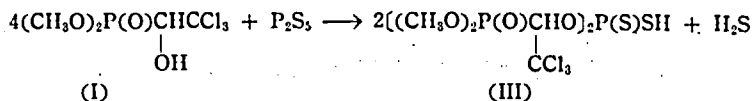
1960-11-30

URL:

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five phosphorus compounds, one of which coincided with Dipterex in Rf. The main product was purified by column chromatography and the barium salt was prepared. By the microanalytical data of barium salt the main product of this reaction



than Dipterex. The compounds (III), when sprayed directly to the insects, was less active than Dipterex. However, when the product was contacted with the insects after passing through the plants, it showed the same activity as that of Dipterex.

This difference on the activity seems to be very interesting. The activities towards the insects and the acetylcholinesterase of brain of housefly will be reported in other papers.

Experimental

A solution suspending 37g (M/7) of Dipterex and 8.9g (M/28) of phosphorus pentasulfide in 80 ml of toluene was heated at 80° for 2 hours under stirring.

The phosphorus pentasulfide became to dissolve in toluene with evolution of hydrogen sulfide, which was led into the aqueous solution of the mercuric chloride to give 2.1g of mercuric sulfide. After the phosphorus pentasulfide dissolved completely, the reaction mixture was continued to stir for one hour at the same temperature. Toluene was distilled off and 42g of the pale yellow viscous oil was obtained. This viscous oil had the characteristic odour of phosphorodithioate and showed to be acidic in aqueous alcoholic solution and soluble in aqueous alkaline solution. Moreover, the viscous oil showed the violet color reaction with cobalt sulfate, reacted with pyridine to give the more viscous syrup which is soluble in alcohol, acetone and water, but insoluble in benzene and ether. By the paper chromatography using butanol, acetic acid and water mixture as solvent (Rf. 0.64 at 25°) and the solution of iodine and chloroplatinic acid as color former, it was clarified that the viscous oil contained one phosphorodithioate. On the other hand, it was shown that the viscous oil contained

was found to be 0,0-bis-1-(0,0-dimethyl phosphonyl)-2,2,2-trichloroethyl phosphorodithioate. Consequently, it was considered that the reaction proceeded according to the following scheme:

The compound (III) was less toxic to the mice

a trace of five phosphorus compounds by using ammonium molybdate as color former. It was determined that one of them was Dipterex in Rf. The pure compound was obtained by passing through the alumina column (3×20cm), using toluene as solvent. Anything could not be detected in the pure compound except one phosphorodithioate.

Into the solution of 27.3g (M/30) of the pure compound in 100 ml of 1-N aqueous solution of barium hydroxide, carbon dioxide was passed to remove the excess barium hydroxide. After the solution was filtered off and concentrated *in vacuo*, 16.8g of white barium salt was obtained and recrystallized from ethanol.

Anal. Calcd. for

$\text{C}_9\text{H}_{14}\text{O}_6\text{Cl}_3\text{P}_2\text{S}_2\frac{1}{2}\text{Ba}$; P, 13.7; S, 9.5

Found P, 13.8; S, 9.3

Summary

The reaction of Dipterex with phosphorus pentasulfide produced neither thiono-Dipterex $(\text{CH}_3\text{O})_2\text{P}(\text{S})\text{CH}(\text{OH})\text{CCl}_3$ nor thiol-Dipterex $(\text{CH}_3\text{O})_2\text{P}(\text{O})\text{CH}(\text{SH})\text{CCl}_3$, but mainly 0,0-bis-1-(0,0-dimethyl phosphonyl)-2,2,2-trichloroethyl phosphorodithioate $[(\text{CH}_3\text{O})_2\text{P}(\text{O})\text{C}(\text{H})(\text{CCl}_3)\text{O}]_2\text{P}(\text{S})\text{SH}$ (III).

Acknowledgement

The authors wish to express their thanks to the Sumitomo Chem. Co. Ltd., for permission to publish this work. They are indebted to co-workers in the analytical section in this laboratory.

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